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new life for an old glass

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High-temperature applications of ceramic materials have been limited as a consequence of their inherently poor thermal shock resistance. However, an exception to this is a very unique glass, vitreous silica (SiO_2), formed by the fusion of rock quartz. When heated to a "bright red" this glass can be quenched in water without fracturing. The excellent thermal shock resistance of vitreous silica can be more easily understood by comparing its coefficient of thermal expansion to those of other glasses. In the temperature range 0-300°C, vitreous silica has an expansion coefficient of 0.5×10^{-6} in/in°C which is 1/7 that of "Pyrex" and 1/20 that of window glass. Some of its present applications are: thermocouple protection tubes for immersion in molten metals, high-temperature laboratory glassware, melting crucibles for semiconductors, and heat-resistant optical windows and lenses.

At approximately 1500°C vitreous silica begins to soften, thus setting an upper temperature limit to its use. However, when used for long periods of time at temperatures even as low as 1200°C, a transformation occurs which converts the amorphous glass into the crystalline phase, Cristobalite. When this occurs, the desirable properties of vitreous silica are lost. Consequently, the long-time, high-temperature use of vitreous silica is not limited by its softening point (1500°C), but by the lower temperature (1200°C) at which crystallization occurs. Thus, there has been a great effort to understand the factors controlling this crystallization phenomenon. From a practical point of view, if the crystallization rate can be reduced, then the satisfactory service life at high temperatures will be increased. This is of great importance since many of the refractory materials employed in rockets and other space applications are in actual use for relatively short periods of time.

It has been observed that crystallization rates vary with different environmental conditions. Through an investigation of atmospheric effects on the crystallization rates of vitreous

silicas of varying histories, a basic difference between the glasses was found. Those vitreous silicas produced under reducing conditions tended to be more resistant to crystallization. As a result of this method of preparation, these glasses were not stoichiometric SiO_2, but had on the average somewhat less than two atoms of oxygen for every atom of silicon. This deficiency of oxygen in the glass inhibits crystallization because the crystalline phase is sensitive to the irregularities which are easily accommodated in the random glass structure. Just how oxygen-deficient this glass can be made and still possess its desirable properties is not known. Work is proceeding in this laboratory to produce very pure vitreous silicas with varying degrees of oxygen deficiency and to measure their crystallization rates under different environmental conditions.

high-pressure apparatus for SEISMIC RESEARCH

Lt. R. E. RIECKER and Lt. K. E. SEIFERT, Terrestrial Sciences Laboratory, AFCRL

A high-pressure, high-temperature shear press, designed to simulate the temperature-pressure environment found 30 to 150 km below the earth's surface, has been delivered to AFCRL. The press, placed in operation last month, will be used to determine the stress and plastic flow properties of rocks under conditions of high hydrostatic pressure and temperature. The objective of these studies is to discover techniques for distinguishing between seismic waves generated by earthquakes and those resulting from underground nuclear explosions.

The shear press is designed to attain simultaneous pressures and temperatures in excess of 750,000 pounds per square inch and 1,000 degrees C, with variable strain rates of from ten revolutions per hour to as little as .01 rph. Major press components include a 300-ton hydraulic jack and a five-kw high-frequency induction heater.

The research program will begin in September with a year's study of olivine. This mineral was chosen because it is believed to be the most abundant mineral in the earth's lower crust. The earth's crust extends to a depth of about 35 to 70 km under land masses. Under oceans, the depth of the crust is about five to ten km. The earth's crust rests on the mantle which makes up more than half of the earth's mass.

Following the study of olivine, three other minerals--enstatite, diopside and labradorite--will be examined. Wafer-shaped samples of minerals used in the press will vary from 1/4 inch to 1 inch in diameter, and will be approximately 1/32 inch thick. After experimentation, the squeezed and heated sample will be examined for structural changes and chemical recombinations. From this work, AFCRL hopes to gain a better understanding of the mechanism of deformation.

The AFCRL experimental rock-deformation program was initiated by Lt. R. E. Riecker and Lt. K. E. Seifert who will also conduct experiments with the press.

crystal growth...the SINE QUA NON of solid state technologies

MAX SWERDLOW Directorate of Physical Sciences, AFOSR

Professor J. J. Gilman, Brown University, whose research on the mechanical behavior of monocrystals is sponsored by AFOSR, writes in the preface of his new book:[1] "Crystals have been admired by man for as long as he has appreciated beauty. The significance of that beauty for a technological society and for the development of scientific knowledge has only begun to be realized, however. The basis of the beauty is now known to be such things as symmetry, structural simplicity, and purity. These characteristics endow crystals with unique physical and chemical properties which have already been used to cause a major transformation of the electronics industry, and systems based on it. Yet to come is the impact of these properties on mechanical technology, on chemical processes, and on the biological sciences. We are passing from a period when solid matter was used mainly in unsophisticated forms into a period when increasing emphasis will be placed on its use in the form of highly organized crystals."

Natural crystals have been used for some time both as gems and for a few special devices such as crystal-controlled oscillators. Now, however, the list of uses for artificial crystals is growing almost daily for such things as frequency-controlled oscillators (quartz); polarizers (calcite, sodium nitrate); transducers (quartz, Rochelle salts, ammonium dihydrogen phosphate); grinding (diamond); radiation detectors (anthracene, potassium chloride); infrared optics (lithium fluoride); bearings (aluminum oxide); transistors (germanium, silicon); magnetic devices (garnets); strain gages (silicon); ultrasonic amplifiers (cadmium sulfide); masers and lasers (ruby, gallium arsenide, calcium tungstate); lenses (fluorite); melting crucibles (magnesium oxide); and tunnel diodes (gallium arsenide). This list is far from exhaustive, but it shows how several old substances (and some new ones) develop unique properties when they have the form of crystals. It seems almost certain that many other substances will reveal unique behaviors when they have also been prepared as crystals. The entire complex of organic chemicals would seem to be especially fertile in this respect, as suggested by the unusual properties of crystallized high polymers.

Developments in the solid-state sciences in the past three decades have had so tremendous an impact on technology that it would take several dozen review papers by specialists to portray a realistic picture of the

1. Gilman, J. J., Editor, The Art and Science of Growing Crystals, John Wiley and Sons, Inc., N.Y., 1963.

breadth and depth of the achievements. There is, however, one fundamental common denominator which pervades the broad expanse of the technological growth. This is the fact that all the progress, regardless of the nature of the specialized device or system which has evolved, has basically resulted from utilization of improved materials. That is, the sine qua non of this tremendous technological growth has been the increased understanding of materials and their properties, the perfection of techniques for improving these properties and, finally, the recognition of unique characteristics and their application to the development of specialized devices. Thus,it is the limitation in our ability to understand and control materials which circumscribes the functions which can be performed by means of a given systems design, and -- perhaps still more important -- the ultimate reliability of that system. This latter consideration becomes increasingly significant as the systems become more and more complex -- as illustrated vividly in our missiles and space programs where the batting average for a perfect functioning of all components is entirely too low.

When artificial crystals were first produced, workers were satisfied with the quality of the product if it had the same symmetry as its natural counterpart. Now they often insist that the crystal have remarkable purity (less than one part per million of impurity). Some crystals have been produced that are not only pure but completely free of such subtle defects as dislocation lines.

It is somewhat ironic that, although artificial crystal growth has had so much effect on various sciences, the process itself has retained more of the flavor of art than of science. This is partly due to a lack of really powerful experimental tools for studying the critical steps of the growth process, and partly due to gradual changes in what is meant by the term, "crystal." An example of the effect of the lack of experimental tools is the fact that the crucial role of surface diffusion rates has been proven only recently. It has long been known that surface diffusion is one step in the growth process; but there was no demonstration that this is often the critical step.

Some years ago, W. G. Pfann[2] described a rather unusual zone-melting technique (Figure 1) which he named "Temperature Gradient Zone Melting (TGZM)." This technique differed from the other members of the zone-melting family in the size of the molten zone, the solute content of the zone, and the driving force used for zone motion. The zones are

2. Pfann, W. G., Zone Melting, John Wiley & Sons, Inc., N.Y., 1957

usually very small (of the order of mils) and may be of slab, cylindrical or spherical shape. They generally have a very high solute content so that they may be molten at temperatures several hundred degrees below the melting point of the solid through which they travel. The zones may be caused to migrate through the solid under the influence of either a temperature difference or a potential difference imposed across the solid. The process is one of dissolution at one face or portion of the zone, solvent and solute transport across the zone, and deposition

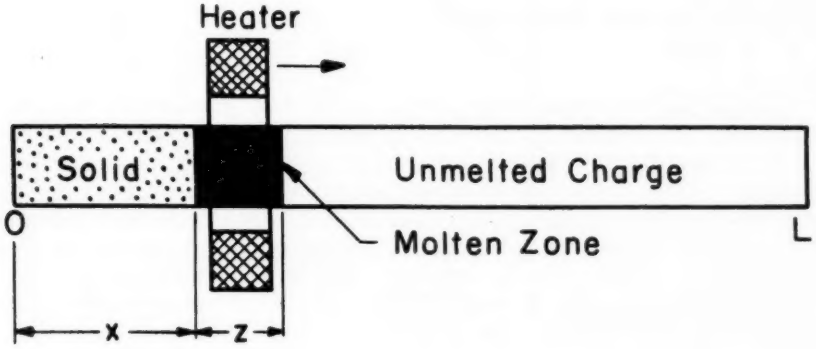

FIGURE 1 – SCHEMATIC ILLUSTRATION OF ZONE MELTING

In this procedure, only part of the charge (ingot or crystal) is melted at any one time, and both melting and freezing are carried out progressively. This simple change in procedure from the normal freezehas made the freezing process more useful and versatile, and offers certain practical advantages in solute control over normal freezing methods. The process variables in this case are (a) zone length, Z; (b) charge length, L; (c) initial distribution of solute in the charge; (d) vapor pressure; (e) mixing conditions, and (f) zone travel rate. By maintaining (a)–(f) constant throughout a zone passage, a variety of different solute distributions will result in the solid for different starting conditions. Further, if these variables are changed during the zone passage, an almost limitless array of solute distributions may be produced.

at the other face or portion of the zone. In the early days of zone melting, this technique was used for the fabrication of n-p-n junctions in transistors of unique shapes, joining, the measurement of liquid diffusivities and for the growth of crystals.

This technique has also been called "The Traveling Solvent Method (TSM)" and the "Liquid-Zone Migration Method (LZM)" by later authors. However, whether it be called TGZM, TSM, or LZM, it has been used for the growth of Ge, Si, GaAs, -SiC, GaP and Al_2O_3 crystals at temperatures of 500^0C to 1000^0C below their melting points, using monocrystalline seeds. Indications are that the regrown material produced by the pas-

sage of the molten zone exhibits a high degree of perfection provided the orientation of the seed crystal is properly chosen.

Interest in the technique seemed to wane in the late 1950's; however, the early 1960's brought about a strong revival of interest in its application to the growth of crystals. The use of a very thin sheet zone has a number of advantages: (1) the apparatus is simple, has no moving parts and the temperatures or potentials need not be altered; (2) the volume of liquid is so small that a container is unnecessary, and contamination therefrom is avoided; (3) the chance of spontaneous nucleation is minimized by the smallness of the zone and the absence of stirring or convection effects.

Supplemental advantages are: (1) the degree of perfection of the crystal due to thermally induced plastic deformation is enhanced since, at the lower-growth temperature, the yield strength of the material is increased; (2) the degree of perfection of the crystal is also enhanced due to the reduction of equilibrium-point defect clusters, since the concentration of these defects decreases exponentially with decrease of the growth temperature; and (3) possible contamination is reduced as the growth temperature decreases and can be eliminated by total enclosure of the sheet zone within the solid.

The prime disadvantages of the technique are: (1) a suitable alloy liquid must be found which does not produce unsuitable doping or alloying in the crystal; (2) the process is slow; and, (3) crystals larger in size than one c.c. require excessively long times.

Motivated by the thought that the migration of a liquid zone through a solid under either a thermal-field or an electric-field driving force can be developed into a very potent technique for both the preparation of crystals and the determination of fundamental information, Dr. W. A. Tiller[3] at the Westinghouse Research and Development Center has, under AFOSR sponsorship, carried out a theoretical analysis of the process. He found that the migration rate of a molten zone through a block of material generally depends upon crystallographic orientation-dependent atomic kinetics of the melting and freezing processes (Figure 2). The magnitude of the atomic kinetic effect depends upon the size of the molten zone, and this size dependence of the mi-

3. Tiller, W. A. "Principles of Solidification," Chapter 15, The Art and Science of Growing Crystals, op.cit.

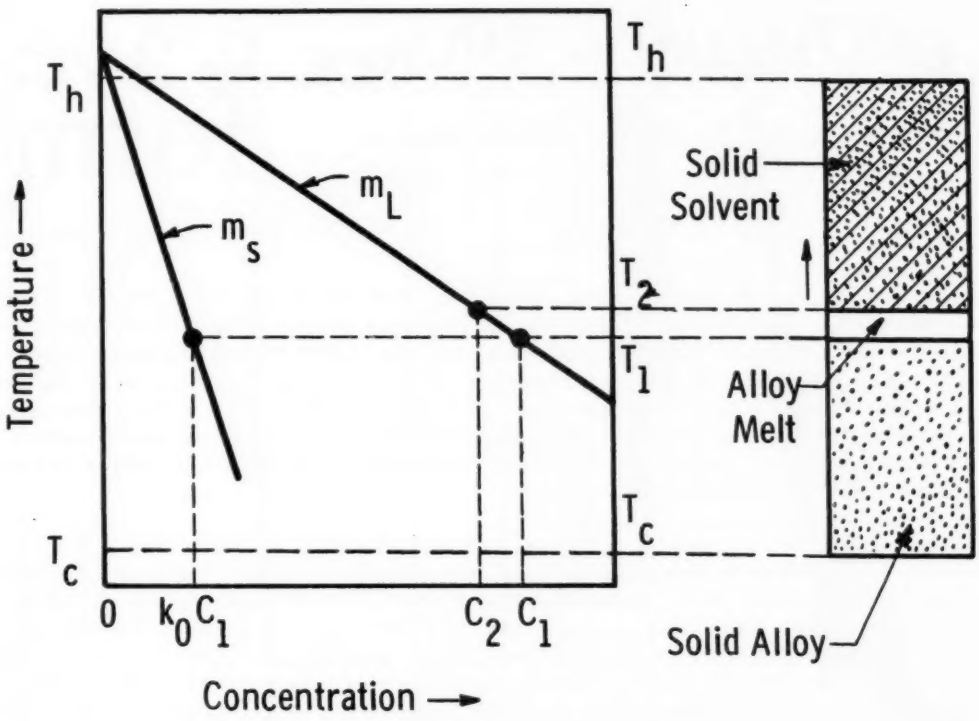

FIGURE 2 – THE MIGRATION OF A LIQUID ZONE THROUGH A SOLID

The starting geometrical and thermal configurations for a slab zone in a block of solid may be as illustrated. The upper portion of the block consists of solid solvent and perhaps some solute. The zone region consists of a substantial alloy of solute and solvent, and the lower block usually consists of a single crystal seed of the solvent. The ensemble is placed in a temperature gradient such that the temperature of the zone region is above the lowest melting temperature, T_c, of the system, and such that the highest temperature, T_h, in the system is below the melting point of the upper or lower blocks. Under these conditions, the zone region melts and the zone adjusts its length until the zone concentration is between C_1 and C_2. Reactions will occur at the two solid – liquid interfaces to produce the equilibrium concentrations C_1 and C_2 at T_1 and T_2 respectively. A solvent concentration gradient is set up in the zone and the combination of solution reaction at T_2, diffusion in the zone and freezing at T_1 causes the molten layer to migrate through the block.

gration velocity may be used as a method for studying the atomic kinetics of alloy melting and freezing. The migration velocity is found to be directly proportional to the temperature gradient in the system. Stable zone migration occurs only for migration in certain crystallographic orientations in a particular system. The effect of an applied electric field is such as to cause the zone to move either faster or slower up the temperature gradient and can, in fact, cause the zone to migrate down the temperature gradient. The analysis agrees with the published experimental data on this technique, thus making it very potent as a technique for both the determination of fundamental information as well as for the preparation of nearly perfect single cyrstals.

EARTHQUAKE-SOURCE wave form

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H. Lamb in his 1904 classical treatment of wave theory, "The Propagation of Tremors Over the Surface of an Elastic Solid," calculated the theoretical form of the surface radial displacement-time pattern produced by the application of a vertical impulse at a point in the surface of a homogeneous half-space. For almost 50 years after Lamb's prediction this pattern had not been observed on earthquake seismograms, although it had been produced in ultrasonic model seismometry in the early 1950's.

Professor Hugo Benioff of the California Institute of Technology's Seismological Laboratory has recently reported on the results of a shallow, very large earthquake in Montana that was recorded on a strain seismograph of his design. This instrument, located in a mine tunnel near Isabella, California, consists of two fuzed quartz rods eighty feet in length, set at right angles to each other. Capable of recording the most minute strains, the instrument recorded the Montana earthquake in almost the exact form predicted by Lamb.

Lamb's prediction was based on a homogeneous half-space, while the earth in the upper portion in fact departs substantially from this, and is multilayered with velocity and density characteristics that vary with depth and azimuth. As a result of these departures from uniformity, the components of a pulse having wave lengths less than the dimensions of the inhomogeneities are reflected, refracted and transformed during propagation in such a way as to produce the complex oscillatory wave train observed on conventional seismograms. While, for small earthquakes, the pulse contains principally high-frequency components which produce oscillatory movements only, the large Montana earthquake had a pulse duration sufficient to include substantial frequency components with wave lengths long in comparison with the crustal inhomogeneities. For these the medium conforms reasonably well to Lamb's condition and consequently produces a wave pattern which approaches Lamb's form. This wave form is produced below, where the Lamb pulse has only been redrafted with the amplitudes of the P (longitudinal body), S (traverse body) and R (Raleigh) waves multiplied respectively by the reciprocals of their velocity and with a reduced time scale to conform more nearly with the amplitude time scale of the earthquake record.

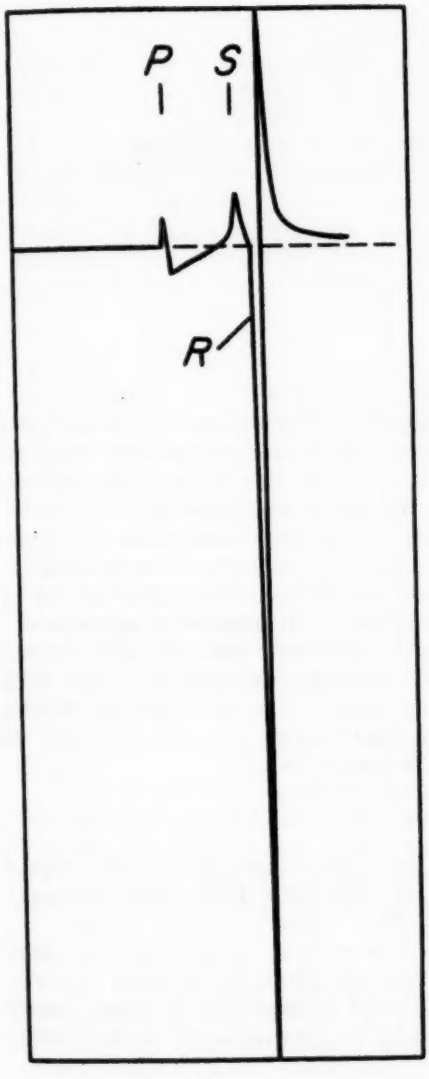

FIGURE 1

LAMB'S PULSE

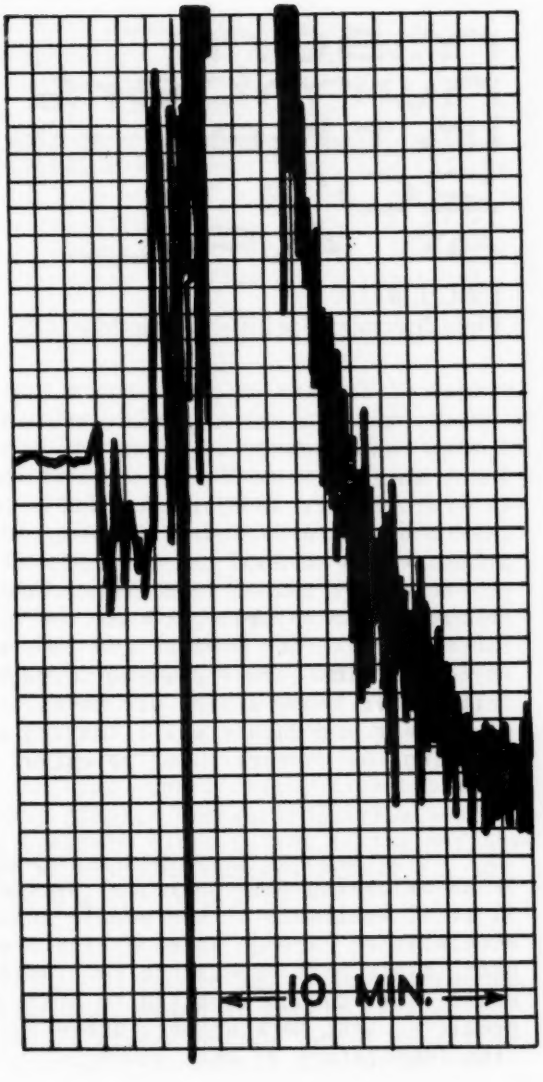

FIGURE 2

SEISMOGRAM FROM
MONTANA EARTHQUAKE

Dr. Benioff's study is sponsored by AFOSR as part of its basic-research effort in support of the Advanced Research Projects Agency's Vela-Uniform program.

mach numbers in the 20's at ARL

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This is not a gay history of numbers in the year 1920 and after, but an announcement of a new facility and its characteristics. The 30-inch, nominal Mach-20, hypersonic wind tunnel of the Aerospace Research Laboratories is in operation and presently being calibrated. This tunnel has been designed for temperatures up to 4400° Rankine (R) and pressures up to 3000 psi upstream of the nozzle throat. A zirconia pebble bed is heated to a temperature not exceeding the above value by combustion of natural gas, air and oxygen under atmospheric pressure. The heater is then pressurized to a chosen pressure and the air heated in counterflow before passing through the hypersonic nozzle. The high initial air temperatures are necessary to avoid liquefaction or freezing of the air components while cooling to temperatures between 20° and 40° R during the conversion of potential to kinetic energy.

The highest temperature to which materials can be heated safely in an oxidizing atmosphere, at present, is 4400°R. This temperature, is still available at the nozzle intake, does theoretically allow acceleration through an expansion process to a Mach number of 18. However, since past experience has indicated that liquefaction in hypersonic flows can be expected to start at lower temperatures than theory predicts, test Mach numbers above 18 should be possible with a stagnation temperature of only 4400°R.

For the facility under discussion, a nozzle for Mach number 18 has been installed for initial calibrations. With this nozzle, which incidentally has a conical wall, Mach numbers between approximately 19 and 21 have been achieved, depending on the initial temperature of the air and gradual changes with time in the diameter of the throat. These changes result from a combination of yielding and deterioration of the nozzle material due to the high temperature and pressure conditions. The higher Mach numbers are achieved with the original throat dimensions and lower stagnation temperatures.

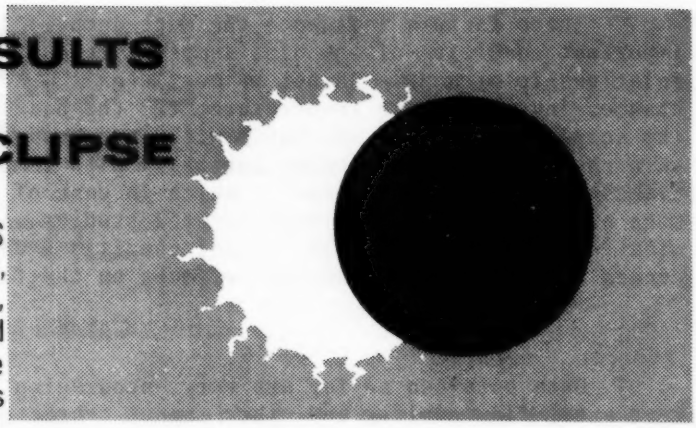
Three factors influence the Mach number: (1) the calorically imperfect condition of the high-temperature gas, changing the area relationship of nozzle throat to nozzle exit, whereby the Mach number changes inversely with temperature; (2) the boundary-layer growth along the nozzle wall, which increases with increasing temperature, thereby also changing the Mach number inversely with temperature; and (3) the liquefaction of air components, which in case of occurrence raises the Mach number. The present experimental studies will lead to a determination of precisely those initial conditions under which liquefaction occurs as well as the numerical effects on the flow characteristics.

Data obtained so far are very encouraging in respect to usable test area, flow quality and other tunnel characteristics. The diameter of the test area in which the variation in Mach number does not exceed $\pm$ 1% has been found to be 20 to 22 inches. The duration of the individual runs lies between 30 and 45 seconds, this being a function of the presently existing vacuum capacity. In the very near future this capacity will be increased by a factor close to 4, thereby increasing the running times by the same factor. The duration of the runs was found to be relatively independent of the stagnation pressure. Maximum total temperature realized to date has been $2960^{\circ}R$ at the nozzle intake and, during the initial phases, stagnation pressures were held below 2000 psi.

Of primary concern in the initial tunnel calibration has been the heater performance, Mach-number (impact pressure)distribution across the test flow, and static pressure distribution in the diffuser. The present testing is being done at Reynolds numbers around 2×10^5. The design Reynold number range is 5×10^4 to 3×10^5.

The present method of heating the pebble bed by combustion has the great disadvantage of producing H_2O, which is partly trapped in the insulation and mixes with the air stream during tunnel operation. Experiments have revealed that the first two tunnel runs after the heating period are producing data of doubtful quality. To correct this situation it is contemplated to replace the combustion heater by an arc-heating device. This new heating method will not only eliminate humidification of the tunnel air, but will also decrease deterioration of the ceramic insulation of the heater and air passages which causes much headache in present-day hypersonic wind-tunnel operation.

SOME RESULTS of the SOLAR ECLIPSE



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Seldom has an event stimulated such widespread and common interest among so many AFCRL research groups as has the solar eclipse of 20 July 1963. This intense interest stemmed from the fact that the eclipse had a bearing on propagation, ionospheric physics, solar and radio astronomy, and atmospheric processes, all of which represent major AFCRL research programs. During the eclipse about 50 scientists took scores of individual measurements from 11 separate locations in Maine, Massachusetts, Canada, and from a KC-135 aircraft. In addition, many AFCRL contractors participated; and two F-104 aircraft flights were arranged by AFCRL for non-AFCRL scientists.

Measurements made by AFCRL scientists were for the most part radio measurements. Therefore, the fact that considerable cloud cover existed on the day of the eclipse didn't affect this aspect of the eclipse program. Only three AFCRL programs, all requiring visual sightings, were affected by this cloud cover.

AFCRL radio experiments were concerned with ionospheric absorption at various frequencies, with residual radio energies from the sun during the eclipse, with the structure of the D, E and F-1 and F-2 layers, and with electron densities in the total ionosphere.

Following are a few of the results:

Using a 10-foot antenna at Hermon, Maine it was possible to compare the residual solar radio energies during the 20 July eclipse with those of the eclipses of 2 October 1959 and 15 February 1961. What makes these comparisons significant is the fact that the 1959 eclipse occurred during an active sunspot period, whereas the 1963 eclipse occurred during a relatively quiet period. At 3100 mcps, the residual signal was 11 per cent during the total eclipse as compared with 20 per cent during the 1959 eclipse. The residual signal results from the fact that the solar diameter at radio wave lengths is greater than at

optical wave lengths; the corona is the main source of signal at frequencies lower than 3000 mcps. Thus, even at totality, solar energy has a residual value.

One of the most interesting new results came from observations at 8 cycles per second. Signals at this frequency increase in strength at night. The reason for this nightime increase is that the space between the earth and the lower ionosphere acts as a resonant cavity. When this cavity is enlarged, due to the increased height of the lower ionosphere, the resonant properties change. The size of the space, of course, determinesthe resonating frequency. Surprisingly enough, during the very brief period of the eclipse, the strength of the 8-cps signal increased to its nighttime level and then decreased rapidly to daytime levels after the eclipse.

Four sites were used to measure the effects of the eclipse on the earth's magnetic field. These sites were located at Weston and Concord, Massachusetts, where about 94 per cent totality was observed, and at Ripley and Orono, Maine, where the total eclipse was observed. In comparing results at the Ripley and Orono sites, it was found that the magnetic-field intensity at Ripley decreased by about 10 gamma, which was twice the decrease observed at the Weston site.

The analysis of all the data collected by AFCRL scientists at the various AFCRL operating sites will continue for many months. The proper correlation of these data and their interpretation, together with the interpretation of data taken by other researchers may result in a much better understanding of solar-terrestrial relationships.

AFCRL notes

LA VERNE E. WOODS
Research Information Office
AFCRL

Three new vocoders, representing a substantial advance over any previously designed equipment of this type, were recently delivered to AFCRL. Vocoders are pieces of equipment used for translating electrical speech signals into digital form. By converting speech signals to digital form, considerable savings in the band width needed to transmit speech is achieved. The improvements in the vocoder equipment grew out of research carried out at AFCRL over the past several years. These forms of equipment can provide digital speech communication at data

rates as low as 1200 bits per second, half the data rate of previous vocoders. Significant betterment has also been achieved in the quality and naturalness of the vocoder speech through improvements in the design of the speech synthesizer.

These pieces of equipment have demonstrated that it is not necessary to have high precision in the vocoder pitch signal in order to obtain natural quality, as had been believed previously. The speech synthesizer is the critical factor. (DATA SCIENCES LABORATORY)

four AFCRL-instrumented rockets were launched in July:

An ionospheric probe, sent up on 2 July, was designed to investigate electrical properties of the lower ionosphere, including ion and electron densities. An Aerobee rocket, flown from the Eglin Gulf Test Range, Florida, carried the instruments to an altitude of 122 miles.

On 9 July, AFCRL used the White Sands Missile Range to launch an Aerobee rocket with a pay load for measuring incident solar radiation from 250 to 1300 angstroms in the extreme ultraviolet portion of the spectrum. A biaxial pointing control was utilized to aim sensing elements at the sun. The rocket carried its 205-pound pay load to an altitude of about 140 miles.

Fort Churchill, Canada, was the site for the launching of a Canadian Black Brant rocket used by AFCRL during the recent 20 July eclipse of the sun to study the ionosphere. Investigations were made in the D and lower E regions at an altitude of 78 miles.

On 18 July, a three-stage research-sounding vehicle, consisting of an Honest John, Nike Booster and Yardbird, carried a pay load of 100 pounds to about 278 nautical miles from the Eglin Gulf Test Range. The flight's objectives were to measure radio propagation parameters below, in and above the F-layer and between the vehicle and the ground, and to flight-test prototype experimental instrumentation to be flown later on a satellite. (Space Physics Laboratory and Upper Atmosphere Physics Laboratory)

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